Table 2

Ex No	Ester substituent	Route	α, β ratio	%Y	MP (°C)
2.1	Benzoyl	3A	4% α, 96% β	100	79
2.2	Benzoyl	3B	82% α, 18% β	93	85
2.3	Ethanoyl	3C	38% α, 62% β	94	77
2.4	Ethanoyl	3A	59% α, 41%β	98	87
2.5	Ethanoyl	3B	95% α, 5% β	86	102
2.6	Benzoyl	3A	5% α, 95% β	100	85
2.7	2-Naphthoyl	3A	3% α, 97% β	100	84
2.8	2-Naphthoyl	3B	94% α, 6% β	100	80
2.9	4-Biphenoyl	3A	8% α, 92% β	100	82
2.10	Cyclohexanoyl	3A	5% α, 95% β	99	77
2.11	Hexanoyl	3A	28% α, 72% β	100	80
2.12	n-Hexadecanoyl	3A	17% α, 83% β	100	65
2.13	n-Hexadecanoyl	3B	97% α, 3% β	100	52

Cellobiose octanonanoate, cellobiose octadecanoate and cellobiose heptanonaoate reference materials, which are employed in Examples 5 to 17 hereinbelow.

Table 3

Ref	Acyl Groups	α, β ratio	%Y	MP (°C)
REF1	Nonanoyl	100% α,	100	97
REF2	Nonanoyl	88% α, 12% β	98	80
REF3	Nonanoyl	1% α, 99% β	100	80
REF4	Decanoyl	85% α, 15%β	84	85
REF5	Nonanoyl	50% α, 50% β	0	114
REF6	Decanoyl	50% α, 50% β	0	105

Example 3

In this Example, further esters were made comprising cellobiose heptanonanoate and a different ester group at the anomeric position. The process comprised a variation of the three stage process described in Example 1 above, the principal differences being indicated herein.

In stage 1, a base catalyst was employed, producing β-D-cellobiose octanonanoate.

D-(+)cellobiose (ACROS; 99% HPLC; 67 % β anomer), nonanoyl acid chloride (83g, 0.47 mol), 96% ALDRICH, (assay; GC: 97.4% with remainder of 2-methyloctanoyl chloride); dry chloroform 99+% (Sure Seal™ bottle, reagent grade, ALDRICH), dry pyridine 99.8+% (sure seal bottle, reagent grade ALDRICH), tetrahydrofuran (Fisher, reagent grade) and methanol (FISHER, reagent grade) were used as received.

A 250 ml three round bottom flask was fitted with a double surface condenser, a pressure equalising funnel and an over head stirrer. All glassware had previously been dried over night at 105°C.

Clear, very faintly yellow nonanoyl chloride (83.2g, 0.47 mol) was added dropwise slowly, taking between 15 and 30 minutes with constant stirring to a solution of cellobiose (10g, 0.029 mol) in dry chloroform (40 mls) and dry pyridine (20 mls) at 50°C. The resultant heterogeneous pale yellow mixture was allowed to react for at 50°C under inert atmosphere and vigorous stirring until monitoring via proton NMR and HPLC indicated that no hepta-substituted cellobiose was present, a period of about 20 hours.

The heterogeneous light yellow mixture was cooled down to room temperature. Then, the reaction mixture was poured into methanol (1000 mls) and stirred for about 15 minutes, producing a precipitate which was recoved by filtration, washed with 50 mls fresh methanol and dried in a vacuum oven (0.8 mbar, 80 Pa) at 40 to 45°C for 5 hours,. 39 g of a white solid was recovered.

The white solid was recrystallised three times from a tetrahydrofuran/methanol mixture (75/200mls), filtered, washed with 50 ml of methanol and dried in a vacuum oven for 5 hours at 40-45°C. 18 g of a white solid (42%) was obtained.